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MEASUREMENT OF INTERNAL RESIDUAL STRAIN GRADIENTS IN METAL MATRIX COMPOSITES USING SYNCHROTRON RADIATION

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INTRODUCTION

Low density titanium based metal matrices combined with high strength and stiffness ceramic fibers are under widespread investigation for possible use in high temperature aerospace applications [1]. Inherent in the processing of these composite materials are the residual stresses resulting from the thermal expansion coefficient mismatch between the fiber and matrix. A tensile hoop and a compressive radial stress result upon cooling from the consolidation temperature because the fiber has a smaller expansion coefficient [2]. The thermal stresses can be very large and may exceed the matrix yield or even fracture stress [3].

X-ray diffraction has been widely used to measure residual stress (actually strain) states at and near free surfaces [4]. However, the traditional techniques are not useful for measuring the strains deep within a composite sample, particularly the rapidly varying strain fields around a small (140μm diameter) fiber. This is because conventionally generated x-rays are strongly absorbed by most engineering materials, penetrating only a few microns.

Efforts to overcome this limitation have included surface measurements, followed by the mechanical or electrochemical removal of a thin layer of surface material, after which another measurement is made [5]. In this way, insight into the strains as a function of depth can be obtained. Unfortunately, the internal strains are altered by the removal of surface layers, and the method is, of course, destructive.

Review of Progress in Quantitative Nondestructive Evaluation, Vol. 11
Edited by D.O. Thompson and D.E. Chimenti, Plenum Press, New York, 1992

Neutron diffraction has been used for residual stress measurement, taking advantage of the high penetration of neutrons in engineering materials [6]. While an internal strain is measured, large diffracting volumes are required because available neutron sources provide relatively low intensities, limiting spatial resolution. Modeling approaches have also been used extensively to analytically predict the sign and magnitude of residual thermal stresses [7]. Experimental validation of the accuracy of these models is urgently needed, since it is unclear to what extent plasticity, creep and fiber/matrix reactions relax or enhance the internal strains.

Energy dispersive diffractometry using high intensity synchrotron radiation offers both good penetration, through several millimeters of titanium, for example, and potentially high spatial resolution. The work reported here explores the feasibility of using this technique for residual strain measurement in a metal matrix composite. The intent has been to measure diffraction from volumes much smaller than the reinforcing fiber, and to compare the measured strains with a simple elastic model.

MEASUREMENT PRINCIPLE

Energy dispersive diffractometry, based on Bragg diffraction, differs from wavelength dispersive methods in that a solid state detector analyzes the distribution of diffracted x-ray energies from a polychromatic (white radiation) incident beam. The detector is positioned at a fixed scattering angle 2θ so that the diffracted energies are obtained by rewriting the Bragg equation as:

$$E_{hkl} = h\nu = \frac{hc}{\lambda} = \frac{hc}{2d_{hkl}\sin\theta} \qquad (1)$$

where h is Plank's constant, ν is frequency and c is the speed of light. The subscripts on E and d indicate specific crystallographic indices. For E given in KeV and d in Å, the above equation becomes:

$$E_{hkl} = \frac{6.22}{d_{hkl}\sin\theta} \qquad (2)$$

For a known constant scattering angle 2θ, each crystallographic orientation with lattice spacing d_{hkl} diffracts at a particular energy E_{hkl}. Measurement of the peak position at this diffracted energy allows the calculation of lattice spacing, from which strain can be determined. The strain thus measured is given by:

$$\varepsilon = \left(\frac{\Delta d}{d}\right)_{hkl} = \left(\frac{\Delta E}{E}\right)_{hkl} \qquad (3)$$

where Δd refers to the change in the lattice spacing of the particular planes and the resultant peak shift ΔE.

The intersection of the incident and diffracted beams defines the x-ray probe. In this region, diffraction is measured only from lattice planes which are perpendicular to the diffraction vector K, the bisector of the angle formed by the incident and diffracted beams. These planes alone satisfy the Bragg condition for diffraction at the particular scattering angle being used. In this way, the hoop and radial strains in the matrix can be measured independently by choosing the orientation of the fiber relative to the incident beam, as shown in Figure 1.

In principle, the ability to measure small strains is limited only by the minimum shift in energy that can be measured for a particular diffraction peak. To improve the accuracy of the peak position determination, a curve fitting technique can be used to fit the detector output, which is Gaussian in nature.

A system of slits is employed to collimate both the incident and diffracted beams, allowing precise control of the incident beam cross section, the scattering angle, the dimensions of the x-ray probe, the angular divergences and the location of the diffracting volume relative to the fiber. These parameters control peak position, peak breadth, count rate and resolution in the strain sensitive direction.

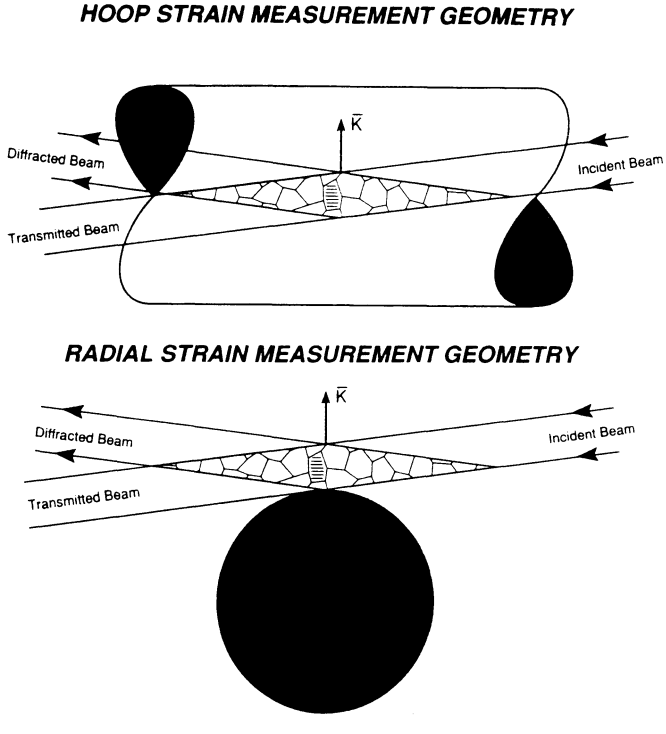

Figure 1. Hoop and radial strain measurement geometries

EXPERIMENTAL PROCEDURE

Model single fiber experimental samples were fabricated by consolidation of either commercially pure titanium powder or an intermetallic Ti-14Al-21Nb (wt%) alloy with either an 800μm diameter alumina fiber or a 142μm diameter silicon carbide SCS-6 fiber by hot isostatic pressing. HIP0 (titanium/alumina) was HIPed at 850°C and 100MPa for 4.0 hours. HIP1 (intermetallic/silicon carbide) was HIPed at 1050°C and 100MPa for 4.0 hours. HIP2 (intermetallic/ silicon carbide) was HIPed at 950°C and 205MPa for 2.0 hours. Samples were controlled cooled at less than 2°C/minute from the hold temperature to 500°C. The resulting sample geometry consisted of a 3-7mm thick flat plate with a single fiber embedded in the plane of the plate at least 2.5mm from any free surface.

All energy dispersive experiments were performed using the NIST X23A3 beamline at the National Synchrotron Light Source, located at Brookhaven National Labs, Long Island, New York. This electron storage ring is 170 meters in circumference and operates at an energy of 2.528 GeV and a current of 200 mA. Usable x-ray energies range from 5 to 60 KeV, with the critical energy at 8 KeV. The beam cross section at the source is roughly 0.15mm X 0.13mm and 20mm X 5mm at the sample.

The x-ray probe volume was determined by a tradeoff with count rate. The intent was always to make the dimensions of the x-ray probe as small as possible, particularly in the strain sensitive direction, so as to avoid averaging over large regions. Typical widths of the incident beam in Figure 1 for example, were 50-100 μm, and the probe volumes used were on the order of 10^{-3} mm^3. The count rates obtained from probe volumes any smaller than this were too low to allow enough points in the sample to be measured within the time constraints.

An EG&G Ortec high purity germanium solid state detector, with a resolution of 190 eV at 5.9 KeV was used to measure the diffracted energy spectrum. An Ortec 92X Spectrum Master provided high bias, amplification and analog to digital conversion. Data acquisition, storage and data analysis was controlled by Ortec Maestro II software operating on a personal computer.

RESULTS and DISCUSSION

Strains were calculated according to equation 3, using the peak position at the point furthest from the fiber/matrix interface as the "strain-free" reference point. Comparison of peak position at each point in the sample with the reference position allows the calculation of strain as a function of distance from the interface.

Figure 2 presents hoop and radial strains as a function of distance from the fiber/matrix interface for the HIP0 sample. The solid line represents a simple elastic model for prediction of the residual strain gradient [8]. Reasonably good agreement is seen between the measured and predicted values.

Several factors were found to influence the degree of scatter in the individual data points, i.e. in the measured peak positions. Among these, counting statistics and time limitations on the availability of the x-ray beam were important considerations. Access to the beamline was limited, and all measurements had to be made during roughly 24 hour periods, between which x-rays were unavailable. Tradeoffs had to be made between the necessity for statistically adequate counts in each peak and the need to make measurements at enough points in the sample to fully characterize the strain condition.

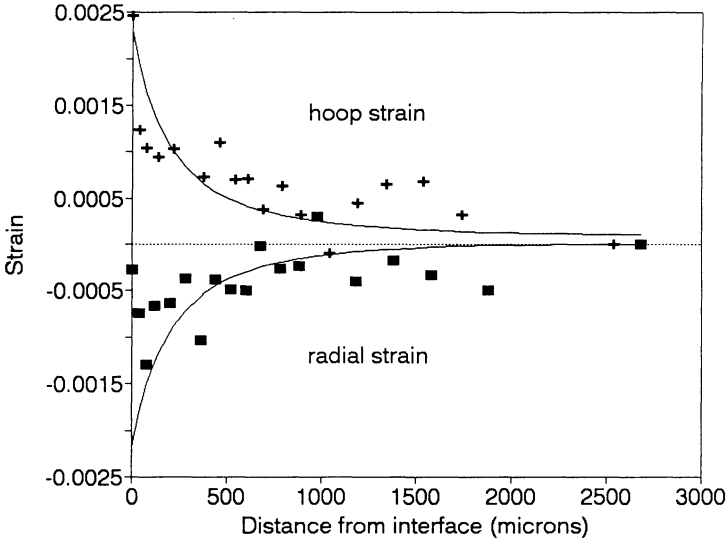

Figure 2. Residual strain profile, HIP0

"Single grain" effects, however, were found to be the primary contributing factor to fluctuations in the data. A basic assumption of the technique is that all of the grains in the probe are scattering at a single angle $2\theta_B$. Properly oriented grains positioned at extremes in the probe can scatter at angles either slightly greater or less than the nominal angle $2\theta_B$, which tends to shift the peak to higher or lower energies. Because of the very small x-ray probes used relative to the 10-20μm matrix grain size, it is believed that individual grains potentially contribute strongly to the peak positions.

Evidence for this was observed experimentally by the fact that the intensity of a particular peak varied greatly at different points in the sample. We calculated that the range of scattering angles possible in a typical probe is large enough to shift a peak equivalent to a strain of 0.001. Presumably, improvements would be made by reducing in the matrix grain size.

Figure 3 shows the hoop strain for the Ti_3Al+Nb/SCS-6 silicon carbide fiber sample (HIP1). We see that near the fiber/matrix interface the strain is lower than expected from the elastic model. Careful examination of the interface (shown in Figure 4) suggests several possible explanations. Radial cracking is observed in the (beta-depleted) zone adjacent to the interface. This serves to relieve strains in this region. The ductile beta phase (white) is absent in this niobium-lean region, leaving only the α_2 ordered hexagonal phase. Niobium is a large atom and its presence dilates the lattice. Its lower concentration provides a mechanism for a small lattice relaxation. Another feature which may influence the residual strain state is the reaction zone, formed of various carbides and silicides with densities different than either the matrix or the fiber [9]. Composition gradients extend long distances from the interface, particularly of interstitials. Using diffusivity data for interstitial diffusion in titanium, we expect carbon and oxygen to diffuse several hundred microns from the interface into the matrix. They would dilate the lattice far from the interface and further depress the apparent interface strain. The amounts of interstitials present and their influence on our results near the interface at present are not known.

Figure 3 also shows the radial strain in HIP2. It also is lower near the interface than one would expect. Debonding and/or porosity were observed at this interface, which we believe may also be relieving strains.

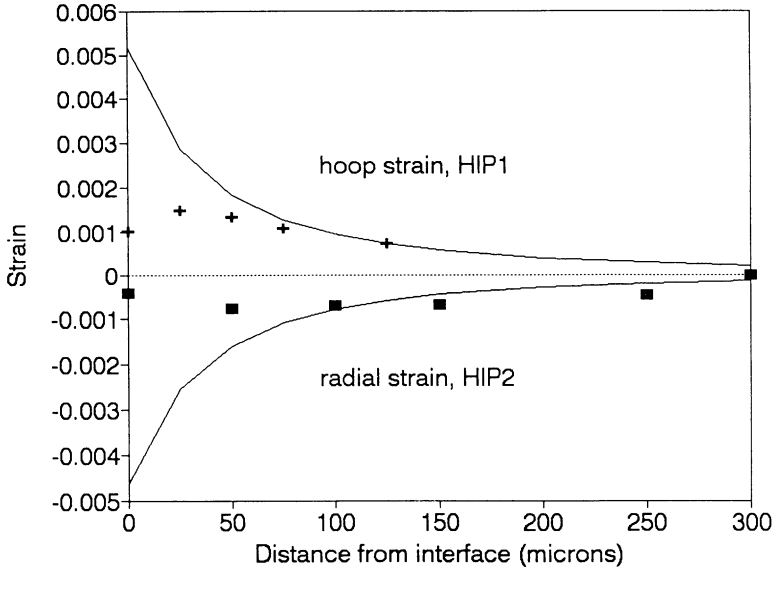

Figure 3. Residual strain profile, HIP1 and HIP2

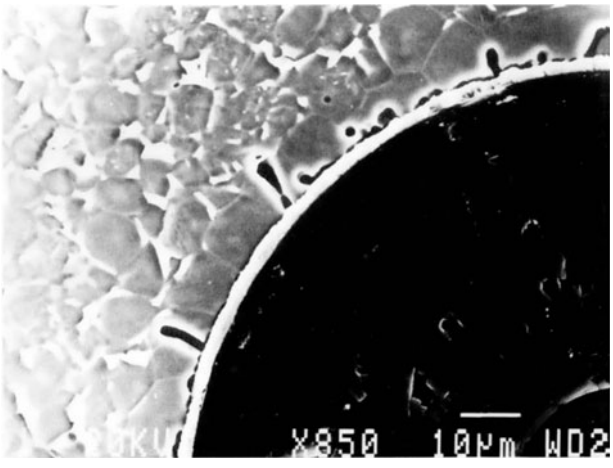

Figure 4. SEM micrograph, HIP1

CONCLUSIONS

The feasibility of using energy dispersive diffractometry to measure thermal residual strains around fibers embedded in HIP consolidated metal matrix composites has been explored. Advantage was taken of the high intensity white radiation available at the National Synchrotron Light Source. The high spatial resolution of the technique was demonstrated by measuring diffraction from volumes on the order of 10^{-3} mm^3.

For the first time, the measurement of thermal residual strain gradients around fibers embedded in a thick composite has been accomplished. Initial results are encouraging, with reasonable agreement between measured and predicted strains for a model composite system. The complexity of the intermetallic/SCS-6 composite system complicated the measurement, and it appears that substantial strain relaxation has occurred close to the interface. The volume of the x-ray probe relative to the sample grain size was found to be the primary contributor to data scatter.

ACKNOWLEDGEMENTS

This work was supported by General Electric Aircraft Engines Division through a Hightemp grant administered by Alex Vary of NASA Lewis. Additional funding was provided by the Academic Enhancement Program of the University of Virginia. We are grateful to P.K. Wright, contract monitor, for his discussions and suggestions throughout the work. Dr. L.M. Hsiung of the University of Virginia has been of great assistance in material characterization and we are grateful for his help.

DISCLAIMER

Information about equipment suppliers is given for completeness and should not be considered an endorsement of their products.

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